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(54) **POLYETHYLENE RESIN COMPOSITION AND FILM MADE THEREFROM**

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a composition desirable for a pressure-sensitive adhesive layer capable of giving a pallet stretch film which possesses pressure-sensitive self-adhesiveness, has good pressure-sensitive adhesiveness between films, can be easily peeled and has good opening natures by mixing a polyethylene resin having a specified composition with a process oil.

SOLUTION: The polyethylene resin used is one having a melt flow rate of 0.1-100 g/10 min, a density of 0.86-0.94 and a Q value of 4 or below. It is produced by copolymerizing ethylene with an α -olefin (e.g. propylene). The process oil used is a paraffinic oil having a weight-average molecular weight of 200-2,000 and is desirably one in which the number of the paraffin chain carbons accounts for at least 50 wt.% of the total number of carbon atoms and which has a kinematic viscosity of 20-800 cSt, a pour point of -40 to 0°C and a flash point of 200-400°C. The mixing ratio is such that 99-80 wt.% polyethylene is mixed with 1-20 wt.% process oil.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] The adhesion reinforcement when this invention having self-adhesiveness and sticking films is good, and when removing, it separates easily, and the opening nature of a film is related with the good polyethylene system resin constituent for films. It is related with the film which consists of a suitable polyethylene resin constituent especially for the adhesive layer of a pallet stretch film, and this constituent.

[0002]

[Description of the Prior Art] the layer and the middle class by whom the typical lamination of a pallet stretch film blended the binder with the copolymer (it is only hereafter written as "EVA".) of ethylene and vinyl acetate in inside and an outer layer -- a line -- it is 3 lamination using low density polyethylene (it is only hereafter written as "LLDPE"). For example, although the both ends of a film are cut off and a film is wound around a paper tube in the above-mentioned multilayer tubular blown film shaping by dividing into two, the cut-off film is blended with LLDPE after re-pelletizing, is fed into the hopper of the making machine for interlayers as it is, and manufactures a film. Here, if a playback pellet is mixed with LLDPE, the reinforcement of a film will fall about 20%. This is because EVA mixes in a playback pellet. Then, a film not using EVA, i.e., the multilayer film all whose layers are LLDPE(s), is desired.

[0003] However, if the conventional LLDPE is used for an adhesive layer, adhesiveness will be inadequate and practical use will not be borne. Although manufacture of the polyethylene of a low consistency was recently attained using the metallocene catalyst etc., if a consistency is made low, adhesion reinforcement will go up, but it blocks at the time of film shaping, and since a film does not carry out opening, practical use is not borne. Moreover, although the binder is blended with the adhesive layer, liquefied polybutene with current [expensive], sorbitan oleate, diglycerol diolate, etc. are used, and the unit price of a film becomes high. These expensive binders are not used but development of the adhesive layer which has target adhesiveness is desired.

[0004]

[Problem(s) to be Solved by the Invention] The adhesion reinforcement of films is good, and when removing, it is going to separate easily, and an expensive binder is not used for the purpose of this invention, but it has self-adhesiveness, and the opening nature of a film tends to offer further the film which consists of the good suitable polyethylene resin constituent especially for the adhesive layer of a pallet stretch film and this good constituent.

[0005]

[Means for Solving the Problem] The adhesion reinforcement when this invention persons not using an expensive binder, but having self-adhesiveness, and sticking films is good. Moreover, by separating easily, when removing, and making the polyethylene system resin which has specific description contain process oil at a specific rate, as a result of repeating research wholeheartedly about a means for the opening nature of a film to obtain a good film Knowledge that the purpose of above-mentioned this invention may be attained is acquired, and it comes to complete this invention.

[0006] That is, this invention is a polyethylene system resin constituent characterized by containing 99 - 80 % of the weight, and process oil for polyethylene system resin at 1 - 20% of the weight of a rate, and is a film with which the consistency which the melt flow rate of polyethylene system resin divides 0.1-100g / 10 is 0.86 - 0.94 g/cm³, and consists of this constituent.

[0007]

[Embodiment of the Invention] Even if it uses it as a simple substance film, the engine performance can be demonstrated enough, but the polyethylene system resin constituent by this invention will demonstrate the function which was excellent much more as a pallet stretch film also from fields, such as tear reinforcement, if the polyethylene system resin constituent by this invention is used as a multilayer film which consists of a layer made into the shape of a film, and other polyethylene resin layers.

[0008] here -- " -- since -- it becomes -- " -- ** -- that to which the resin layer according [saying] to this invention exists in an innermost layer or the outermost layer -- In this case, the other layers are other polyethylene resin layers, may reach and may recognize variety existence. [two or more] Moreover, also in that to which the resin layer by this invention exists in an innermost layer and the outermost layer, and this case, the other layers are other polyethylene resin layers, and mean plurality and that variety existence may be recognized. In addition, as long as the purpose of this invention is attained, the resin layer and other polyethylene resin layers by this invention do not need to be prepared all over the film, therefore this invention contains such a film.

[0009] Below, each component, process, and film of this invention are explained.

1. The polyethylene system resin constituent by polyethylene system resin this invention is characterized by containing polyethylene system resin and process oil at a specific rate, and it is [polyethylene system resin] desirable to use what shows the description and the physical properties of following (1), (2), and (3).

[0010] (1) the melt flow rate (MFR: melting flow rate) according [the polyethylene system resin used in this invention] to JIS-K7210 -- 0.1-100g/-- 0.3-80g / thing that shows the physical properties for 0.5-50g / 10 minutes more preferably is used preferably for 10 minutes for 10 minutes. If this MFR is larger than the above-mentioned range, since thermal resistance and film reinforcement will fall and membrane formation of a film will become unstable, it is not desirable. Moreover, since the resin pressure force will become high and extrusion nature will fall if this MFR is smaller than the above-mentioned range, it is not desirable.

[0011] (2) 0.86 - 0.94 g/cm³ and the thing which shows 0.865 - 0.935 g/cm³ preferably are used for the consistency according [the polyethylene system resin used in this invention] to JIS-K7112. If this consistency is larger than the above-mentioned range, since self-adhesiveness will become poor and the adhesion reinforcement of films will fall, it is not desirable. Moreover, if this consistency is too smaller than the above-mentioned range, since films will block and films will not separate, it is not desirable.

[0012] (3) 2.7 or less thing is especially used for the Q value (weight average molecular weight/number average molecular weight) asked for the polyethylene system resin used in this invention by steric exclusion chromatography (Size Exclusion Chromatography: SEC) preferably three or less four or less. If this Q value exceeds the above-mentioned range, since a dry area, self-adhesiveness, and the adhesiveness of films will fall [a film front face], it is not desirable.

[0013] 2. The polyethylene system resin used in process this invention of polyethylene system resin is ethylene and the copolymer of an alpha olefin, and there is especially no constraint about the catalyst and polymerization method in manufacture of this copolymer. As a catalyst, a Ziegler type catalyst (namely, thing based on the combination of support or a non-supporting halogen content titanium compound, and an organoaluminium compound), the Philips mold catalyst (namely, thing based on support chrome oxide (Cr⁶⁺)), and the Kaminsky mold catalyst (namely, support or a non-supporting metallocene compound and an organoaluminium compound, especially the thing based on the combination of alumoxane) are mentioned, for example. As a polymerization method, 200kg/cm² or more and polymerization temperature are mentioned [a high-pressure bulk polymerization method 130 degrees C or more etc.] for the slurry method under existence of these catalysts, a gaseous-phase

fluidized bed process (for example, approach given in JP,59-23011,A) and a solution method, or a pressure.

[0014] Since it is desirable to have comparatively narrow molecular weight distribution, as for the polyethylene system resin in this invention, it is desirable to use especially the Kaminsky mold catalyst. As the concrete manufacture approach, JP,58-19309,A, JP,59-95292,A, JP,60-35005,A, JP,60-35006,A, JP,60-35007,A, JP,60-35008,A, JP,60-35009,A, JP,61-130314,A, Each official report of JP,3-163088,A, the European Patent application public presentation No. 420,436 specification, The approach indicated by the U.S. Pat. No. 5,055,438 specification, the international public presentation official report WO 91/No. 04257 specification, etc., Namely, a metallocene catalyst, a metallocene/alumoxane catalyst, Or the catalyst which consists of a compound which reacts with a metallocene compound and a metallocene catalyst which are indicated by the international public presentation official report WO 92/No. 07123 specification, for example, and serves as stable ion is used. The approach of carrying out copolymerization of the ethylene of a principal component and the alpha olefin of the carbon numbers 3-18 of an accessory constituent can be mentioned.

[0015] The alpha olefin used as a comonomer is a 1-olefin of carbon numbers 3-18, and, specifically, is a propylene, 1-butene, 1-pentene, 1-hexene, 1-octene, 1-heptene, the 4-methyl-pentene -1, and a 4-methyl-hexene. - 1, 4, and 4-dimethyl pentene-1 grade can be mentioned. As a comonomer, the above-mentioned alpha olefin is not restricted to one kind, but is contained as what also has the desirable plural system copolymer used two or more kinds like a terpolymer.

[0016] 3. as the process oil used for process oil this invention -- weight average molecular weight -- 200-2000 -- the thing of 300-1500 can be mentioned preferably. It is the mixture which combined three persons of aromatic series, naphthenes, and paraffin, and that to which a paraffin chain carbon number occupies 50 % of the weight or more in total carbon is called paraffin series oil, that whose naphthene ring carbon number is 30 - 45 % of the weight is called naphthene oil, what has more aromatic series carbon numbers than 30 % of the weight is called aromatic series system oil, and such process oil is distinguished. In these, it is more desirable than a heat-resistant point to use paraffin series oil.

[0017] As paraffin series oil used by this invention, 50 - 600cSt and the pour point are used for the kinematic viscosity of 40 degrees C, and -40-0 degree C of 200-400 degrees C of 250-350-degree C oil is preferably used for -30-0 degree C and the flash point (COC) suitably 20 to 800 cSt (centistokes).

[0018] 4. For 99 - 80 % of the weight, and process oil, in polyethylene system resin and blending-ratio-of-coal this invention of process oil, polyethylene system resin is [polyethylene system resin / 98.7 - 83 % of the weight and the process oil of the blending ratio of coal of polyethylene system resin and process oil] 1.3 - 17% of the weight of rates preferably one to 20% of the weight. If the rate of process oil is larger than the above-mentioned range, membranes cannot be formed and it will not become film-like. Moreover, since bridging may be carried out, or a surging may be carried out, it may extrude and it may change under the hopper of an extruder, it is not desirable. Moreover, if the rate of process oil is smaller than the above-mentioned range, since films will not block and separate but they will be lost, it is not desirable. [of self-adhesiveness]

[0019] 5. About the process of the polyethylene system resin constituent which contains the polyethylene system resin and process oil in process this invention of a polyethylene system resin constituent at a specific rate, both can be manufactured by blending polyethylene system resin and process oil according to the manufacture approach of the usual resin constituent, and the same approach. The dryblend of polyethylene system resin and the process oil may be carried out beforehand, and you may specifically supply to the hopper of a film making machine as it is, and using an extruder, the Brabender plastograph, a Banbury mixer, a kneader blender, etc., it can knead, and can consider as a pellet type by melting and the approach usually used, and a film can also be manufactured.

[0020] moreover, polyethylene system resin and the polyethylene system resin constituent containing process oil can blend the auxiliary additive component generally used as an object for resin constituents, for example, an anti-oxidant, (especially, a phenol system and the Lynn system anti-oxidant are desirable), an anti blocking agent, a slipping agent, a thermostabilizer, an ultraviolet ray absorbent, a neutralizer, an antifogger, a coloring agent, an antimicrobial agent, adhesives, etc. moreover, the giant

molecule which consists of a monomer in which other copolymerization is possible with extent by which the effectiveness of this invention is not spoiled to the AUW of polyethylene system resin and the polyethylene system resin constituent containing process oil, for example, high pressure process low density polyethylene, and a line -- low density polyethylene etc. can also be blended five to 30% of the weight.

[0021] 6. The polyethylene system resin constituent by formation this invention of a simple substance film and a multilayer film can be made into the shape of a film, can carry out a laminating to the polyethylene resin of independent or others, and can be used as a pallet stretch film. When carrying out the laminating of the layer of the polyethylene system resin constituent in this invention, and other polyethylene resin layers, formation thru/or the laminating of each class may be the thing of pertinent arbitration. There are an approach of forming each class in the shape of a film separately beforehand, making paste them up after that according to the shaping approach of the conventional multilayer film, and carrying out a laminating, a method of performing formation and the laminating of each class at the same process with an extrusion method, etc. In the case of the former, manufacture of a film can adopt air-cooling inflation molding, a 2 steps of air-cooling cooling tubular film process, T die film shaping, water-cooled inflation molding, etc. Moreover, as a latter ejector system, there are approaches, such as the extrusion laminating method, the dry laminate method, the sandwiches laminating method, and a co-extrusion method (the co-extrusion which does not prepare a glue line, the co-extrusion which prepares a glue line, and the co-extrusion which blends adhesion resin are included). In this invention, various kinds of multilayer films can be obtained by any approach.

[0022] The resin layer in which the substitution-gestalt of the pallet stretch film by this invention contains polyethylene system resin and process oil has self-adhesiveness, and is excellent in adhesion reinforcement, and since removing easily is possible when removing, it is the gestalt of the shape of a film prepared in the innermost layer or the outermost layer, the innermost layer, and the outermost layer. In addition, also when making the polyethylene system resin constituent in this invention into the shape of a film alone, the above-mentioned inflation-molding method, the T die film fabricating method, etc. can be adopted.

[0023]

[Example] The following examples and examples of a comparison are for explaining this invention still more concretely. The measurement of physical properties and film physical-properties evaluation in these examples and the example of a comparison are a thing when carrying out by the approach shown below.

[0024] 1. It is Based on Measuring Method (1) MFR:JIS-K7210 of Physical Properties (190 Degrees C, 2.16Kg Load).

(2) consistency: -- JIS-K7112 -- conformity (3) Q-value: -- measure by the Measuring condition shown below using steric exclusion chromatography (Size ExclusionChromatography:SEC), and ask from weight average molecular weight and number average molecular weight. In addition, with mono dispersion polystyrene, the universal calibration curve was created and it calculated as molecular weight of the polyethylene of a straight chain.

model: -- Waters Model 150C GPC solvent: -- o-dichlorobenzene rate-of-flow: -- a part for 1ml/--

temperature: -- 140-degree-C measurement concentration: -- 2mg [/ml] injection rate: -- 200microl

column: -- the Showa Denko K.K. make -- AD80M S Three [0025] 2. Film Physical-Properties

Evaluation Approach (1) 180-Degree Exfoliation Adhesion Reinforcement (Outline was Illustrated to Drawing 1)

Once cutting down the film (the shape of a film of two sheets to which insides were joined) which formed membranes by inflation molding in a 23-degree C thermostatic chamber in width of face of 100mm, and magnitude with a die length of 200mm and removing the film of two sheets which has adhered, an inside and external surface are doubled and 15kg load is applied for 5 minutes. And 200g load was given at the tip of a smooth plate, and 180-degree exfoliation adhesion reinforcement was measured using the slip circuit tester (HEIDON-14 made from New East Science mold) which the smooth plate moved according to the load at high speed. The above-mentioned film which lapped two

sheet is placed on a smooth plate, a lower film is fixed, an upper film is inserted with a clip, and, specifically, a load cell is connected with a monofilament to a clip. In addition, a clip covers with paper on an upper film, and puts in the paper. And the smooth plate was moved at high speed and the maximum reinforcement in case 180 degrees of films of two sheets exfoliate was made into the 180-degree exfoliation adhesion value on the strength.

[0026] (2) The film (the shape of a film of two sheets to which insides were joined) which formed membranes by 180° blocking peel strength inflation molding was cut down in the 23-degree C thermostatic chamber in width of face of 100mm, and magnitude with a die length of 200mm, and 180-degree blocking peel strength was measured with the slip circuit tester (HEIDON-14 made from New East Science mold). The film (since it fabricates uncivilized) with which insides adhered is placed on a smooth plate, a lower film is fixed, an upper film is inserted with a clip, and, specifically, a load cell is connected with a monofilament to a clip. In addition, like the time of 180-degree-C exfoliation adhesion measurement on the strength, a clip covers with paper on an upper film, and puts in the paper. And the smooth plate was moved the rate for 150mm/, and reinforcement in case 180 degrees of films of two sheets exfoliate was made into 180-degree blocking peel strength value.

[0027] (3) Once cutting down the film (the shape of a film of two sheets to which insides were joined) which formed membranes by self-adhesiveness inflation molding in a 23-degree C thermostatic chamber in width of face of 50mm, and magnitude with a die length of 200mm and removing the film of two sheets which has adhered, place the film of one sheet so that external surface may turn up. It has the film of one more sheet so that an inside may turn down, and it puts on the placed film (the external surface and inside of a film stick). At this time, a film is not pressed down using a finger etc. And when films adhered, O and films considered as time of ***** or inside ** x. Self-adhesiveness O means that self-adhesiveness is in the film, and self-adhesiveness x means that there is no self-adhesiveness in the film.

[0028] The polyethylene system resin used in the example and the example of a comparison, process oil, and the film shaping approach are as follows.

(a) Preparation of the synthetic catalyst of polyethylene system resin was carried out by the approach indicated by JP,61-130314,A. namely, complex ethylene screw (4, 5, 6, 7-tetrahydro indenyl) zirconium dichloride 2.0 millimol -- methyl AMMOKISAN by the Oriental Stow fur company -- the above-mentioned complex -- receiving -- a 1000-mol time -- in addition, it diluted with toluene to 10l., the catalyst solution was prepared, and the polymerization was performed by the following approaches.

[0029] The mixture of ethylene and 1-hexene was supplied so that the presentation of 1-hexene might become 83 % of the weight, the pressure in a reactor was kept at 1300kg/cm², it reacted to the churning type autoclave mold successive reaction machine of 1.5l. of content volume at the temperature of 115 degrees C, and 0.895 g/cm³ and Q value obtained the polyethylene system resin whose MFRs are the ethylene and 1-hexene copolymer 3.5g / 10 minutes, and whose consistency are 3.1 (henceforth PE-1). Moreover, except having changed the presentation of 1-hexene at the time of a polymerization, and polymerization temperature, catalyst preparation and a polymerization were performed by the same approach as the above, and 0.880 g/cm³ and Q value obtained the polyethylene system resin whose MFRs are the ethylene and 1-hexene copolymer 3.5g / 10 minutes, and whose consistency are 3.3 (henceforth PE-2).

[0030] (b) Process oil by (process oil i) Idemitsu Kosan Diana process PW-90[paraffin-series oil, average molecular weight: 539 or 40-degree C kinematic viscosity:95.5cSt] (henceforth PW-90) and process oil by (ii) Idemitsu Kosan Diana process PW-380[paraffin-series oil, average molecular weight: Kinematic viscosity [of 746 or 40 degrees C]:381.6cSt] (henceforth PW-380) was used.

[0031] (c) Air-cooling tubular blown film shaping was performed for film shaping polyethylene system resin and process oil on condition that the following.

a <process condition> -- model: -- a Tommy air-cooling tubular blown film making machine Diameter of screw;40mmphi ratio-of-length-to-diameter;24 Temperature; 130 degrees C Diameter of die;75mmphi Die lip; 3mm Dice temperature; 130 degrees C Blow ratio; 2.1 Taking over rate; a part for 9m/ Film thickness; 50micro [0032] PE-1 was blended as example 1 polyethylene system resin, PW-90 were blended with 98 % of the weight 2% of the weight as process oil, after carrying out dryblend, it

supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1.

[0033] PE-1 was blended as example 2 polyethylene system resin, PW-90 were blended with 96 % of the weight 4% of the weight as process oil, after carrying out dryblend, it supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1.

[0034] After blending and carrying out the dryblend of PE-2 as process oil as an example 3 - 5 polyethylene system resin at a rate which shows PW-90 in Table 1, it supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1.

[0035] After blending and carrying out the dryblend of PE-2 as process oil as an example 6 and 7 polyethylene system resin at a rate which shows PW-380 in Table 1, it supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1.

[0036] PE-2 of example of comparison 1 polyethylene system resin were supplied to the hopper of an air-cooling tubular blown film making machine as they were, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 2. Although 180-degree exfoliation adhesion reinforcement is excellent, its 180-degree blocking peel strength is large, and it is very inferior in the opening nature of a film. Moreover, self-adhesiveness is also inferior.

[0037] PE-2 were blended as example of comparison 2 polyethylene system resin, PW-90 were blended with 99.5 % of the weight 0.5% of the weight as process oil, after carrying out dryblend, it supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1. Although 180-degree exfoliation adhesion reinforcement is excellent, its 180-degree blocking peel strength is large, and it is very inferior in the opening nature of a film. Moreover, self-adhesiveness is also inferior.

[0038] Membranes were not able to be formed, although PE-2 were blended as example of comparison 3 polyethylene system resin, and PW-90 were blended with 75 % of the weight 25% of the weight as process oil, and it supplied to the hopper of an air-cooling tubular blown film making machine as it is after carrying out dryblend.

[0039] PE-2 were blended as example of comparison 4 polyethylene system resin, PW-380 were blended with 99.5 % of the weight 0.5% of the weight as process oil, after carrying out dryblend, it supplied to the hopper of an air-cooling tubular blown film making machine as it is, the film was manufactured, and the film was evaluated. The result of evaluation is as being shown in Table 1. Although 180-degree exfoliation adhesion reinforcement is excellent, its 180-degree blocking peel strength is large, and it is very inferior in the opening nature of a film. Moreover, self-adhesiveness is also inferior.

[0040] Membranes were not able to be formed, although PE-2 were blended as example of comparison 5 polyethylene system resin, and PW-380 were blended with 75 % of the weight 25% of the weight as process oil, and it supplied to the hopper of an air-cooling tubular blown film making machine as it is after carrying out dryblend.

[0041]

[Table 1]

		単位	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7
ポリエチレン系樹脂	MFR	g/10min	3.5	3.5	3.5	3.5	3.5	3.5	3.5
	密度	g/cm ³	0.895	0.895	0.880	0.880	0.880	0.880	0.880
	Q値	-	3.1	3.1	3.3	3.3	3.3	3.3	3.3
	割合	%	88	86	88	84	85	86	84
プロセスオイル	PW-80	%	2	4	2	6	15	-	-
	PW-380	%	-	-	-	-	-	4	6
180° 剥離粘着強度		g	52	21	304	271	63	269	275
180° ブロッキング剥離強度		g	5.1	3.9	22	14	5.4	15	11
自己粘着性		-	○	○	○	○	○	○	○

[0042]

[Table 2]

		単位	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5
ポリエチレン系樹脂	MFR	g/10min	3.5	3.5	3.5	3.5	3.5
	密度	g/cm ³	0.880	0.880	0.880	0.880	0.880
	Q値	-	3.3	3.3	3.3	3.3	3.3
	割合	%	100	99.5	75	99.5	75
プロセスオイル	PW-80	%	-	0.5	25	-	-
	PW-380	%	-	-	-	0.5	25
180° 剥離粘着強度		g	302	300	-	297	-
180° ブロッキング剥離強度		g	68	60	-	56	-
自己粘着性		-	×	×	-	×	-

[0043]

[Effect of the Invention] According to this invention, the adhesion reinforcement when sticking films is good, and when removing, it can separate easily, and an expensive binder is not used, but there is self-adhesiveness and the opening nature of a film can obtain the suitable film for the adhesive layer of a good film, especially a pallet stretch film.

[Translation done.]